# MEASURING DISLODGEABLE PROPARGITE RESIDUE ON FIELD-GROWN ROSES AND PENETRATED CLOTHING RESIDUES

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#### **ABSTRACT**

Propargite is used as an acaracide on a variety of crops including fieldgrown roses. An analysis of the dissipation profile for dislodgeable foliar residues on field-grown roses was performed in Kern County, California, in Following aerial application at customary application rates (1.5 lb of active ingredient/acre), foliage samples were taken at selected time intervals. In addition, whole leaf samples were also collected to establish a surface area to weight relationship for future whole-leaf sampling methods. Mean foliar residue levels on the third day after application were  $2.21 \text{ ug/cm}^2$ ; on the seventh day  $2.14 \text{ ug/cm}^2$ ; on the fourteenth day 0.92ug/cm<sup>2</sup>; and on the twenty-first day 0.16 ug/cm<sup>2</sup>. Dosimetery clothing was also worn under normal work clothing by the sampling personnel as a surrogate upper-bound indication of exposure during cultural practices in roses. There were residues on the dermal dosimeters, ranging from 0.628 mg/hour (day of application) to 0.196 mg/hr (Day 7) to 0.042 mg/hr (Day 11). Dosimetery results were not reflective of normal cultural practice, since samplers entered densely foliated fields which are normally not entered until after mechanical defoliation. Worker dermal exposure in rose production is reduced by use of the chemically resistant chaps, multiple layers of clothing and heavy leather gloves normally worn in this physically hazardous (thorns) environment.

#### INTRODUCTION

In October 1988, propargite (OMITER, COMITER) was made a restricted-use material with longer reentry intervals and greater safety requirements than previously required. This was primarily in response to propargite being identified as a potential reproductive toxin. One of the crops affected by the new reentry intervals was roses. The reentry interval was raised to 21 days post-application. This was a default interval in that no valid data existed on roses to substantiate a shorter reentry interval. This interval is in stark contrast to the 3 day interval on strawberries. strawberries and roses are in the same phylogenetic family (Rosaceae), grower representatives had argued that the 21 day interval is too long and should be closer to the strawberry interval, which was set using actual foliar residue data. This study was designed to develop a foliar residue data-base for propargite on roses in order to arrive at a scientifically valid reentry interval. A study of the surface-area to leaf-weight relationship was also conducted.

In addition to foliage analysis, an effort was made to establish the potential for residue penetration through protective equipment. Dosimetery clothing was to be used to estimate exposure to workers entering the treated fields at critical time periods.

#### METHODS AND MATERIALS

Cooperation of the Bear Creek Production Co. (Wasco, CA) was secured. This company is the major producer of field-grown roses in California. As part of their normal agricultural practices, OMITER 30W is applied to the roses to supress mite populations. The sampling sites designated as Field 902 and Field 502 were "mother block" fields. These plants are grown for root stock, and as such are allowed to grow tall (up to 2 meters) and untrained. Field 502 was especially dense, with very small leaves. These fields are seldom, if ever, entered by fieldworkers but did have considerable foliage for sampling and worker contact. The sites designated Field 101 and Field 701 were the better trained, second year stock which grew to 1 meter or less.

#### **Foliage**

Three fields (sampling sites) were selected for sampling. One of the fields was large enough to divide (70 acres), allowing for a fourth site to be created. On the day before the applications, foliar samples were collected, using a variation on the Gunther <u>et al</u>. method (1) to establish preapplication residue levels, since some of the fields had undergone previous applications.

Leafpunch foliage samples were collected using either a  $1.75~\rm cm~(2.405~\rm cm^2$  area) or  $1.25~\rm cm~(1.227~\rm cm^2$  area) diameter BIRKESTRAND leafpunch. A total of either 60 leaf discs, from the larger punch, or 100 discs from the smaller, were generated per 4 ounce sample bottle. The smaller diameter leafpunch was used for the smaller leaves (Field 502). Each field had 4 sampling sites, one in each quadrant of the field (NE,NW,SE,SW). Each sampling site had one sample (bottle) taken from it. The sampling sites were defined by the planting rows. The person doing the sampling (the "sampler") would walk approximately 10 meters into the field within the

sampled row. The sampler would then take a leafpunch sample in a pattern dependent on the number/size of discs taken. For 60 discs, the sampler would punch two leaves on the right, two on the left, and then move on about 0.5 meter to the next subsite of leaves to sample. For 100 disc sampling sites, because of the dense nature of the foliage and its small leaf size, the sampler would take 10 punches at each subsite and then move on 1 or more meters. Each sampled row was marked with surveyor tape to prevent resampling of a row which had already been physically disturbed by the sampler. The punches were cleaned with distilled water and disposable towels between sampling sites.

The sampling schedule for leafpunching was pre-application, Day 0 (day of application), 1, 2, 3, 4, 7, 8, 10, 11, 14, 15, and 21 days after application.

Tared sample bottles (16 oz) were used to collect whole-leaf samples for propargite analysis. Samples were collected from opposing quadrants (either NE+SW or NW+SE). Leaves were cut at the petiole as it joined the blade, taking care to minimize contact. In all cases only healthy leaves were selected with no obvious insect or fungal damage. Thirty leaves were collected in each container. Because of equipment limitations, whole-leaf samples were collected from Fields 101, 701 and 902 on Day 0, Day 1 and Day 2 only.

On the day of application, a 250 ml sample of tank mix was obtained. The application rate was 5 pounds of OMITE<sup>R</sup> 30W per acre (5.61 kg/hectare) which is equivalent to 1.5 pounds of propargite per acre (1.69 kg/hectare). The pesticide was diluted into 20 gallons of water per acre (187 liters/hectare). One pint per 100 gallons of an unspecified spreadersticker was used. A sample of the formulated product was also obtained to check for adequacy of formulation (sufficient active ingredient).

All bottle samples were sealed with aluminum foil (food grade) and capped with screw-on plastic or steel caps. The samples were kept on ice and in a FREEZE-SAFER or other thermally insulated container. The samples were shipped to CDFA Sacramento Chemistry Laboratory Services for analysis (Appendix B). The dislodgeable residue results were reported as  $ug/cm^2$ . Tank mix was reported as mg/L and dry formulation was reported as percentage of weight. All raw data is retained in the Worker Health and Safety Branch.

In two fields (902 and 101), fifteen additional bottles were collected (total of 30) for measurement of physical parameters, both gravimetrically and on the leaf surface-area meter. Each bottle had a specific number of leaves, between 5 and 75, inclusive, incrementing in units of five. The samples were collected in tared bottles, stored on ice and weighed within 4 hours. These samples were not subject to chemical analysis. The field weighing unit was an OHAUS Triple-beam Balance (Model #700). The balance was calibrated before each series of measurements using Troemner Calibration Weights (VWR Scientific, traceable to NBS). Bottles were wiped dry before measuring.

After weighing, these samples were again stored on ice and sent to Sacramento for measurement on a photometric surface area meter (LI-3100 Area Meter, LiCor, Inc). Samples were measured twice, with the average used for calculations. Error between each sample measurement was <1 percent.

## Exposure Dosimetry

The samplers were dosimeter clothing during Days 0, 1, 7, 8, 10, 11, 14, 15, and 21. Dosimeter clothing included upper-body, 100 percent cotton long-sleeved T-shirts, cotton gloves (Day 0 only), and cotton-synthetic blend tights (Day 21 only). These dosimeters were worn underneath the normal protective clothing. The shirts were under the coveralls, the cotton gloves under the leather work gloves and the tights under both coveralls and vinyl chaps. The dosimeters were worn during the time required to drive to the field and conduct the sampling. The T-shirt dosimeters were dissected into sleeve and torso components, and all dosimeters were placed into plastic storage bags (ZIPLOCK brand). These samples were stored on dry ice. The length of time the dosimeters were worn was noted. Analysis was performed by Chemistry Laboratory Services.

The leather work gloves worn by the samplers throughout the study were also collected at the end of the study for analysis of aqueous surfactant dislodgeable propargite. The gloves were randomly distributed amongst the samplers at the start of each sampling day. The gloves were stored with all the other sampling/protective equipment and no effort was made to prevent cross-contamination with boots, coveralls or chaps, although coveralls were laundered each time prior to wearing them.

#### RESULTS

Table One shows the gravimetric/surface area measurements of the whole-leaf samples. Series I were taken on Day 0 of the study and Series II were taken on Day 21.

Table Two shows the results of the dislodgeable foliar residue samples taken using a leafpunch. The mean and standard deviation of the four quadrants in each field is given. On Day Seven, the grower reapplied propargite to field 902, using the same application parameters. According to the grower, mite control was not obtained by the first application. Figures One through Four show graphs of the degradation for each field, Figure Five is a compilation of Fields 502, 101, 701 and the period of the first application for 902. In this compilation graph, data from Field 502/Day 1, Field 101/Day 4 and Field 902/Days 7-21 were not included (as outliers) for being unusually high, unusually low and from a second application, respectively. Figure Six combines Fields 101 and 701, since these two fields were created by dividing the larger, single field.

Table Three presents the dermal dosimetery results. In all cases where dermal dosimeters were used, there was always an upper-body dosimeter. In a few of the situations, as noted, either gloves or lower-body dosimeters were also employed.

Analysis of the formulation demonstrated that the product was not exactly at the percent of the labeled concentration (30.0 percent active ingredient) but it was within California's acceptable minimum of 28.6 percent (actual propargite content 28.84 percent by weight). The application mixture, taken directly from the aircraft, was 11.6 g/L of propargite. This is very close to the expected concentration of 10 g/L, derived from the reported inputs of 758 grams of propargite in 75.8 liters of water.

The four pairs of leather gloves had a mean propargite residue level of  $9.89 \pm 1.47$  mg.

Whole-leaf data are presented in Table Four. Whole leaves were collected during the first three days after application (Day 0 to Day 2). The results were in good agreement with the leafpunch-derived samples (absolute mean daily percent difference = 14.4%). Leaf surface area was estimated using power curve fitting ( $r^2 = 0.99$ ) and solving for the equation of (surface area) = 95 (leaf weight)1.01. No samples were collected from Field 502 since the leaves were very small and not representative of average rose foliage size.

TABLE ONE: Leaf number to weight to surface area relationship.

				· · · · · · · · · · · · · · · · · · ·
	SEI	RIES I	SERI	IES II
NUMBER OF <u>LEAVES</u>	LEAF <u>WEIGHT</u>	SURFACE <u>AREA</u>	LEAF <u>WEIGHT</u>	SURFACE <u>AREA</u>
5 10 15 20 25 30 35 40 45 50 55 60 65 70	0.68 g 2.59 g 2.17 g 6.79 g 6.35 g 7.30 g 8.70 g 12.20 g 13.68 g 10.44 g 10.36 g 15.61 g 11.22 g 10.77 g	67 cm <sup>2</sup> 206 cm <sup>2</sup> 180 cm <sup>2</sup> 554 cm <sup>2</sup> 539 cm <sup>2</sup> 654 cm <sup>2</sup> 765 cm <sup>2</sup> 1,078 cm <sup>2</sup> 1,170 cm <sup>2</sup> 937 cm <sup>2</sup> 901 cm <sup>2</sup> 1,336 cm <sup>2</sup> 946 cm <sup>2</sup> 957 cm <sup>2</sup>	2.69 g 4.62 g 6.93 g 6.81 g 10.69 g 12.29 g 12.28 g 13.90 g 14.65 g 18.10 g 16.02 g 16.88 g	Sample - 245 cm <sup>2</sup> 395 cm <sup>2</sup> 551 cm <sup>2</sup> 607 cm <sup>2</sup> 875 cm <sup>2</sup> 997 cm <sup>2</sup> 1,066 cm <sup>2</sup> 1,147 cm <sup>2</sup> 1,273 cm <sup>2</sup> 1,569 cm <sup>2</sup> 1,415 cm <sup>2</sup> 1,487 cm <sup>2</sup> 1,588 cm <sup>2</sup>
75 	19.73 g	1,623 cm <sup>2</sup>	19.45 g	1,670 cm <sup>2</sup>

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TABLE TWO: Mean dislodgeable propargite residue in each sampled field. Values are in  $ug/cm^2$  and are from leafpunch data (n = 4 for all sampled fields).

		Fiel	d Identific	ation Numbe	e <b>r</b>
		101	701	902	<u>502</u>
Day Pre-Ap	MEAN	0.05	NS	0.12	0.97
Day Fle-Ap	S.D.	0.03	NS	0.15	0.34
Day 0	MEAN	2.43	3.81	1.22	2.40
<i>j</i>	S.D.	0.44	1.39	0.70	1.22
Day 1	MEAN	2.58	4.03	1.03	0.34
•	S.D.	1.25	0.55	0.39	0.09
Day 2	MEAN	2.11	3.78	1.97	3.18
	S.D.	0.34	1.62	0.90	1.17
Day 3	MEAN	1.85	2.35	1.59	3.03
	S.D.	0.51	0.46	0.16	2.17
Day 4	MEAN S.D.	<b>5.83</b> 3.06	<b>2.47</b> 0.72	1.62	1.60
				0.18	1.22
Day 7	MEAN S.D.	1. <b>80</b> 0.92	1. <b>96</b> 0.98	<b>8.60*</b> 1.83	<b>3.14</b> 0.64
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Day 8	MEAN S.D.	1.27 0.67	1.75 0.95	<b>3.69*</b> 0.50	<b>3.68</b> 0.76
D 10	MEAN	0.31			
Day 10	S.D.	0.31	<b>0.33</b> 0.18	<b>2.90*</b> 0.87	3.62 3.89
Day 11	MEAN	2.31	0.76	2.46*	1.84
Day II	S.D.	1.02	0.32	0.75	0.26
Day 14	MEAN	1.83	0.55	1.66*	0.86
	S.D.	0.51	0.35	0.32	0.39
Day 15	MEAN	1.73	0.62	1.78*	0.88
	S.D.	0.67	0.32	0.14	0,53
Day 21	MEAN	0.04	0.22	0.44*	0.23
	S.D.	0.02	0.13	0.20	0.25
Half-Life (d	ays)	18	8.5	6	13

\*reapplication NS - No Sample

TABLE THREE: Dermal dosimeter results (primarily upper-body T-shirts)from sampling crews. Dosimeters were worn under all other protective equipment (coveralls, leather gloves and/or vinyl chaps). Values are in total mg per specific body area.

DAY/	SUBJECT	UPPER	UPPER-BODY	<del></del>	
HOURS EXPOSED		BODY	mg/hour	GLOVES	LOWER-BODY
0	#1	4.66			
5 Hours	#2	4.03		1.08	
	#3	3.11		1.50	
	#4	0.74		0.60	
	MEAN	3.14	0.63	1.06	
1	#1	3.11			
3 Hours	#2	3.80			
	#3	2.13			
	#4	1.25			
	MEAN	2.57	0.85		·
7	#1	0.67			
5 Hours	#2	1.29			
	MEAN	0.98	0.20		
8	#1	0.65			
4 Hours	#2	2.05			
	MEAN	1.35	0.34		
10	#3	0.81			
6 Hours	#5	0.19			
	#6	0.19			
	MEAN	0.40	0.07		
11	#3	0.33			
6 Hours	#5	0.28			
	#6	0.14			
	MEAN	0.25	0.04		
14/15	#1	0.57			
8 Hours	#7	1.32			
	MEAN	0.95	0.12		
21	#1	0.29			0.19
5 Hours	#2	1.03			0.11
	MEAN	0.66	0.13		0.15

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TABLE FOUR: Mean dislodgeable propargite residue levels from whole-leaf samples. Values are in  $ug/cm^2$  (n = 2 for all sampled fields).

		Field Ident	ification Nu	mber
<u>DAY</u>	<u>101</u>	<u>701</u>	<u>902</u>	<u>Daily Mean</u>
0	3,86	3.31	1.51	2.89
1	2.69	4.25	NS	3.47
2	2,43	2.02	NS	2.23

NS-No Sample

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All control dosimeters had non-detectable (<10 ug/sample) levels of propargite. Propargite was present on the foliage on the day before application. The average pre-application levels for each field were 0.05 ug/cm $^2$  for Fields 101 and 701, 0.12 ug/cm $^2$  for Field 902 and 0.97 ug/cm $^2$  for Field 502.

Weather data for the area (Appendix A, California Department of Water Resources, Shafter Area) indicated that no precipitation fell during the course of the study. The average maximum temperature was greater than  $95^{\circ}F$ , the average minimum above  $55^{\circ}F$ . Daily relative humidity ranged from high 70's to mid 30's (percents).

#### DISCUSSION

The gravimetric/surface-area (GSA) power curve analysis supports further investigation into this method for whole-leaf residue assessment. The equation developed from the rose GSA data was used to calculate the foliar residue levels for the whole-leaf samples. The relatively close agreement between the leafpunch residue levels and the whole-leaf levels suggest that the whole-leaf method may be suitable for use by persons who do not have access to leafpunches when required to ascertain foliar residue levels.

Graphical interpolation of the degradation profiles figures for each of the individual fields indicates a range of half-lives, from 6 days (Field 902) to 18 days (Field 101), with an average half-life of 11±5 days. Average foliar residue levels showed a steady decline except for an unexplained rapid decrease (and equally unexplained increase) in Fields 101, 701 and 502 on Day 10. The highest mean foliar residue for the single application fields occured on Day 4 in Field 101: 5.83 ug/cm<sup>2</sup>. However, this value represents a spike in the degradation profile (3X greater than either the preceding or subsequent residue level) and is suspect. The two-application Field 902 had an even greater maximum mean foliar residue level, 8.60 ug/cm<sup>2</sup>, on the first day of the second application.

Whole-leaf data differed slightly from leafpunch data. On Day 0, the whole leaf samples were 16 percent greater than leafpunch; on Day 1 whole leaf was 5 percent higher; on Day 2 whole leaf was 24 percent lower. This method's

variability is no greater than the variability historically found in leafpunch samples collected by this Branch and may be useful for rapid sample collection.

The dermal dosimeter results from the sampling personnel showed that human exposure to propargite residues was not high. On Day 0, the upper-body dosimeters had an average residue value of 3.14 mg. Though no lower-body dosimeters were available for Day 0, there were some available on Day 21. Comparing Day 21's upper-body dosimeters to Day 0's and using this ratio on Day 21's lower-body dosimeters allow for a calculated Day 0 lower-body dosimeter value, which equals 0.714 mg. Adding all the dosimeter values and normalizing to 8 hours exposure results in a daily potential dermal exposure of 7.9 mg/day if a worker were to enter after spray has dried and dust has This, however, is not a feasible reentry time since propargite, settled. aside from its potential for reproductive toxicity, is known to cause chemical dermatitis. Fresh residues may lead to severe dermal irritation. Day 3 has been suggested as a potential reentry time, but no dosimeter data is available for that time interval. There is leaf residue data for both time periods. Calculating a mean transfer factor for days in which there is both dosimeter data and leaf residue data would allow interpolation of the missing dosimeter data. Transfer factors (TF) are derived by dividing the dermal exposure by the dislodgeable foliar residue (DFR), using the method of Zweig, et al. (2). The TF for Day 1, using mean upper-body dosimeter (857 ug/hr) and mean DFR (2.55 ug/cm<sup>2</sup>, n=3) is equal to 336 cm<sup>2</sup>/hr. Likewise, Day 7 has a TF of 85 cm<sup>2</sup>/hr (DFR n=3). By linear interpolation, Day 3 should have a TF of 265 cm<sup>2</sup>/hour. Multiplying by the mean DFR of Day 3 (2.21  $ug/cm^2$ , n=4) gives a product of 585.6 ug/hr for the upper-body dosimeter. Day 0 dosimeter information suggests that 64 percent of the total exposure may be from the upper-body exposure, leaving the remaining 36 percent distributed over the rest of the body. Thus, if 585.6 ug/hr corresponds to the upper-body exposure, the remaining body should be exposed to 329.4 ug/hr for a total of 915 ug/hr or 7.32 mg/day. This estimated dermal exposure level to personnel involved in rose foliage sampling is lower than both grape cane-turners' exposure at 28 days post-application (10.1 mg/day) and peach harvesters' exposure at 21 days post-application (7.6 mg/day)(3).

Using 7.32 mg/day, an Absorbed Daily Dosage (ADD) can be calculated. Propargite dermal absorption has been estimated to be 17 percent/day (3). This absorption rate for a 70 kg male results in an absorbed dosage of 17.8 ug/kg/day ([7.32 mg/day x 0.17] : 70 kg). An analysis of rose production cultural practices suggests that workers may be in contact with treated foliage (budding, weeding, suckering, top breaking) between 30 to 90 days per year. These values assume that the daily exposure will always be 7.32 mg/day (no degradation) and that rose worker exposure in normally cultivated fields is equivalent to sampling personnel exposure in dense, normally unentered fields. Actual rose worker propargite exposure may be much lower.

### REFERENCES

- Gunther, F.A., W.E Westlake, J.H. Barkley, W. Winterlin, L. Langbehn: Establishing dislodgable pesticide residues on leaf surfaces. <u>Bull.</u> <u>Environ. Contam. Toxicol.</u> 4, 243 (1973).
- 2. Zweig, G. and W. Popendorf: J. Agric. Food Chem. 32, 1232 (1984).
- 3. Thongsinthusak, T., J. Ross, J. Sanborn, D. Meinders, H. Fong, D, Haskell, C. Rech, R.I. Krieger: Estimation of exposure of persons in California to pesticide products that contain propargite. CDFA Worker Health and Safety Report HS-1527 (revised Nov. 30, 1989).

Figure 1: Field 101

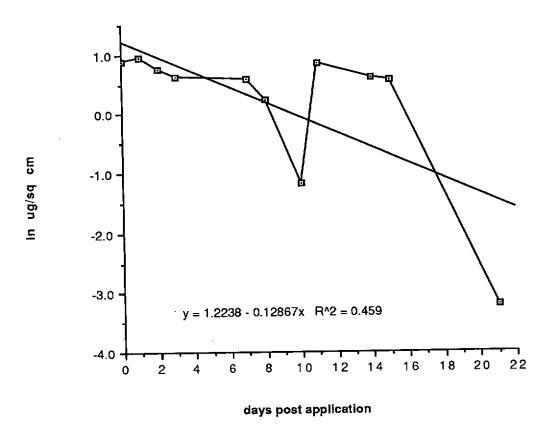


Figure 2: Field 701

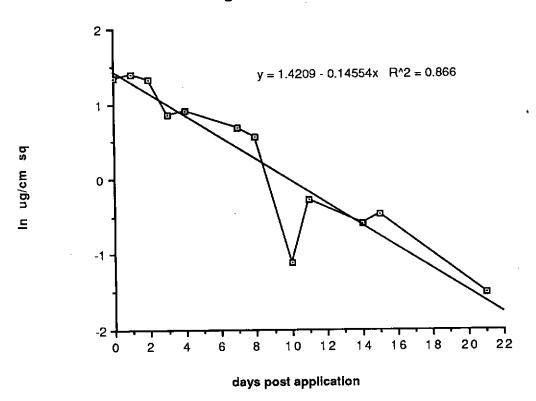


Figure 3: Field 902

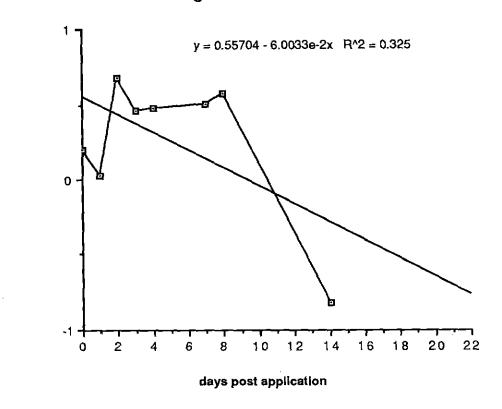


Figure 4: Field 502

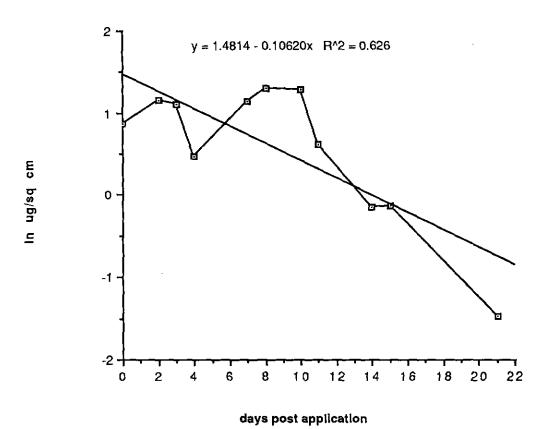


Figure 5: Grand Mean of all Fields

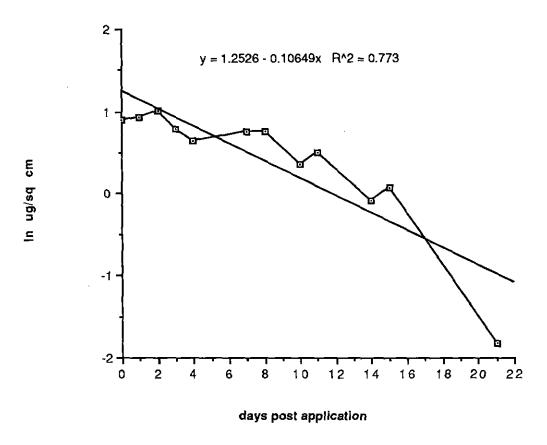
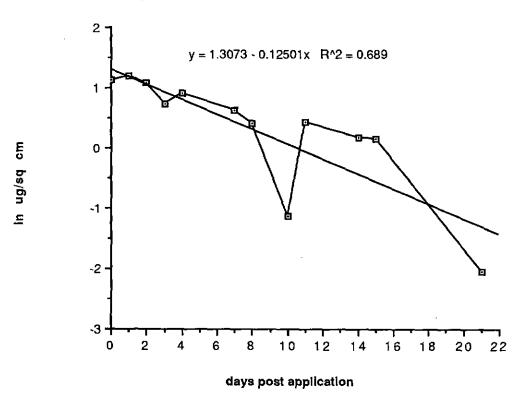


Figure 6: Mean of Fields 101 and 701



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Daily Weather Data for Station # 5

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California Irrigation Management Information System Department of Water Resources Shafter/USDA

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Daily Weather Data for Station # 5

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